3-35-62-6 • AUGUST 1962

TECHNICAL REPORT

INVESTIGATION OF ELECTRONIC MODULE POTTING RESINS

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INVESTIGATION OF ELECTRONIC MODULE POTTING RESINS

by

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WORK CARRIED OUT UNDER AIR FORCE CONTRACT AF 04-647-787

Lockheed

MISSILES & SPACE COMPANY

A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION

SUNNYVALE, CALIFORNIA

FOREWORD

Space Systems Division Value Engineering, 62-81, initiated this study of encapsulant and freeze-coating compounds. The memo and supplement shown in the Appendix were sent to SSD Product Analysis, 64-63, describing the various trial compounds and the amplifier assemblies, or modules, which were to be encapsulated. Engineering Design Techniques, 58-26, was given the assignment of investigating these compounds and their use as encapsulants.

Acknowledgments are expressed to Richard Fullerton, Associate Engineer, for his contribution to this investigation. The work was carried out under the direction of Dr. Hans M. Wagner, Section Head, Processing Techniques, and L. L. Libby, Manager, Engineering Design Techniques.

ABSTRACT

Five PAM amplifier modules (Dwg. No. 1322517) were used as test vehicles in the evaluation of encapsulants and coatings. Three modules were encapsulated, respectively, in one of the following resins:

- Furane Plastics Epocast 202/9615 at 110 phr
- Products Research PR 1538
- Emerson & Cumings Stycast 2651 MM

One module was freeze-coated in Dennis Chemical Co. Insulating Lacquer No. 1162 and then encapsulatedin Epocast 202/9615; another module was freeze-coated in Dow Corning Adhesive 271 and then encapsulated in Stycast 2651 MM. The three resins were measured for shrinkage, insulation resistance, hardness, exotherm and strain. The encapsulation and tests on the resins were performed in the Processing Techniques Section of Engineering Design Techniques, Dept. 58-26.

Space Systems Division Product Analysis, Dept. 64-63, will use thermal-shock environmental tests, in accordance with ATP MO 68118, to determine the suitability of these materials as encapsulants and coatings for the PAM amplifiers modules.

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Section 1
INTRODUCTION

The improvement of encapsulants and potting compounds for electronic devices is required by increasingly rigorous environmental conditions of Space Systems programs. Improved testing methods are also demanded for intelligent evaluation of the thousands of formulations available.

Because of the general lack of adequate data, electronics designers and packaging engineers are often forced to select encapsulants on the basis of their own experience, the experience of others, or the claims of manufacturers. Usually the design or packaging concept is so new that there is little previous experience for reference nor enough time or money available for a testing program for selection of the best encapsulant.

Complete physical and electrical property data on encapsulating and potting resins are needed along with data on the following:

(1) Amount of shrinkage during cure
(2) Exotherm generated during cure
(3) Stress on components during thermal cycling.

The effect of cure temperature on these properties is a variable often overlooked. The presence of heat-sensitive electronic devices in some instances makes it necessary to use curing temperatures lower than those recommended by the resin formulation; in other instances, cure temperatures are increased to speed-up production rates without taking into consideration the effect of increased cure temperatures on exotherm, shrinkage and internal stress. Adequate cure schedules can be determined by such methods as insulation resistance testing during cure.

Resin-shrinkage and thermal cycling stresses can be relieved or counteracted by certain processing techniques described in this report, e.g., the use of freeze-coatings that prevent adhesion or act as cushioning or strain equalizers on delicate components. These items are all discussed in this report by studying not only the encapsulant material characteristics above, but also as applied to the actual encapsulation of electronic modules.

Section 2
EQUIPMENT AND PROCEDURES

2.1 FREEZE-COATING MATERIALS

Listed below are the two resin coating systems that were tested in conjunction with the resin encapsulants.

(1) LAC 37-4070, Class 1 (Dennis Chemical Co. compound No. 1162 A and B), is a solvent solution of a long-chain semiflexible epoxy resin and a room temperature curing agent.

(2) Dow Corning 271 Adhesive is a solvent solution of a pressure-sensitive silicone adhesive which, in this application, functions as a cushioning material and prevents the formation of a tenacious bond between the encapsulating resin and the components.

2.2 ENCAPSULANTS

Resin-encapsulation materials receive the lion's share of study in this investigation. This investigation is also so constructed as to study resins both as a separate entity and as used in encapsulation procedure.

Table 2-1 identifies and describes encapsulants and freeze-coatings. Immediately following this table is another, Table 2-2, which lists a summary of (1) the modules which were encapsulated in resins and (2) the modules which were freeze-coated before encapsulation.

Table 2-1

MATERIAL IDENTIFICATION

Manufacturer	Material Name	Lot No.	Specification No.	Description
Dennis Chemical Co.	No. 1162 Insulation Lacquer	1B-5992 and 1B-5993	LAC 37-4070	Epoxy resin solution
Dow Corning Corp.	Dow Corning 271 Adhesive	1165	–	Silicone resin adhesive
Furane Plastics	Epocast 202/9615	1-8-70 (202) and 1-8-59 (9615)	LAC 40-4093	Unfilled, semi-flexible, epoxy-polyamide resin system
Emerson & Cuming	Stycast 2651 MM	464	LAC 40-4088	Filled, low viscosity, epoxy resin
Products Research	PR 1538	B-x77 Resin and A-R252 Curing Agent	–	Unfilled, medium viscosity, polyurethane resin system

Table 2-2

MODULE ENCAPSULATION SUMMARY

Module No.	Freeze Coat	Encapsulant	Cure Schedule
103	Dennis 1162	Epocast 202/9615	24 hr/77° F
104	None	Epocast 202/9615	3 hr/150° F
105	DC 271	Stycast 2651 MM	2 hr/165° F
108	None	Stycast 2651 MM	2 hr/165° F
08	None	PR 1538	16 hr/150° F

Table 2-3 lists the encapsulation material properties. Note that these data are partially the offering of manufacturers and partially LMSC.

Table 2-3

ENCAPSULATION MATERIAL PROPERTIES

Name of Product Manufacturer Chemical Nature		Epocast 202/9615 at 110 phr[b] Furane Plastics Epoxy – Polyamide	Stycast 2651 MM, Catalyst 11 Emerson & Cuming, Inc. Low Viscosity Epoxy Resin	PR 1538 Products Research Co. Polyurethane
Properties				
Thermal				
1. Thermal Conductivity	cal x cm^{-1} x $°C^{-1}$ x 10^{-4}		5.16	3.16
2. Resistance to Heat	(cont.) °C		205°	300°
3. Heat Distortion Point	°C at 264 psi			
4. Linear Thermal Expansion	10^{-5} x $°C^{-1}$		2.5	24
Physical				
1. Hardness at 20°C	Specify	45 – 52 Shore D[a]	88 Shore D[a]	77 Shore A[a], 24 Shore D[a]
2. Compressive Strength	lb $inch^{-2}$		14,000	
3. Volume Shrinkage	%	3.78[a]	2.38[a]	1.4 0.63[a]
4. Specific Gravity	–	1.06[a]	1.58[a]	1.08[a]
5. Tensile Strength	lb $inch^{-2}$		7,000	3,000
6. Flexural Strength	lb $inch^{-2}$		12,000	
Electrical				
1. Volume Resistivity	ohm – cm	> 2 x 10^{12} at 25°C	4 x 10^{16}	1 x 10^{13}
2. Surface Resistivity	ohms			5 x 10^{12}
3. Dielectric Strength	volts x mil^{-1}		440	275
4. Dielectric Constant	At 60 cps and 10^6 cps	3.5 and 3.0	4.4 at 10^8	4.4 at 10^6
5. Dissipation Factor	At 60 cps and 10^6 cps	.010 and .021	0.02 at 10^8	
Handling				
1. Viscosity at 20°C	Centipoises	10,000	4500	10,000
2. Pot Life – 100 grams	Minutes at °C	120/25°C	4 hr/20°C	60/25°C
3. Typical Cure	Hours at °C	24 hr/25°C or 4 hr/66°C	2 hr/75°C	4/85°C
4. Exotherm – 200 grams 50 grams	Peak temp. °C	See Figures 4-4, 4-5, 4-6 and 4-7	See Figures 4-7 and 4-9	See Figures 4-10 and 4-11

(a) Test Data, LMSC.
(b) phr – parts per hundred resin

2.2.1 Shrinkage During Cure

Theory. Shrinkage of the resin around embedded components during the cure cycle causes internal stresses in the resin. High shrinkage in a resin sets up correspondingly high-stress levels and, with certain resins, the shrinkage is sufficient to cause cracking of the resin.

The two common methods of measuring shrinkage (i.e., change in dimension of a known volume and change in specific gravity of the uncured and cured resin) are actually summations of three separate shrinkage factors. These factors are:

(1) Shrinkage during polymerization while the resin is in the liquid state
(2) Shrinkage after the resin has gelled or hardened
(3) Thermal contraction of the resin during the change from oven or exothermic temperature to room temperature

The first factor, shrinkage in the liquid state, is significant (approximately 50 percent of total shrinkage) but is technically unimportant since the liquid resin cannot exert any stress on embedded components or set up stress concentrations in the resin itself. The shrinkage after gelation or hardening is the technically important shrinkage since it is during this stage that actual stresses are being set up. The total thermal shrinkage of the resin depends on the difference between oven or exothermic temperature, room temperature, and the coefficient of thermal expansion of the particular compound.

A simple test has been devised by Parry and Mackay* to measure the shrinkage of the resin after gelation.

Equipment. Shrinkage test equipment are as follows:

(1) Constant-Temperature Bath
A Fisher constant-temperature bath is used to insure isothermal

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*H. L. Parry and H. A. Mackay, "Cure Shrinkage of Epoxy Systems," J. Soc. Plastics Engrs., Vol. 14, Jul 1958, pp 22-24.

conditions during curing. It is charged with a light mineral oil and equipped with a controlling thermostat and power stirrer.

(2) Dilatometer

A simple dilatometer is fabricated by inserting a 1.0 ml graduated pipette through a cork stopper into a standard 2 oz dropper bottle. The resin under test is placed in the bottle but no release agent is applied to the bottle. This prevents the resin from pulling away from the bottle and creating air pockets.

Procedure. The test procedure is as follows:

(1) Preheat all equipment and materials to test temperature (150° F).
(2) Put 25 gm of degassed resin in a 2 oz dropper bottle and fill the bottle with DC 200 silicone oil.
(3) Fill the pipet with silicone oil and insert into the dropper bottle.
(4) Adjust the oil level in the pipet (the required level is different for each resin system).
(5) Place the bottle in the constant-temperature bath. Another sample is placed in the bath at this time to determine the gel point.
(6) Monitor the oil level and record the oil level in the pipet at the gel point (as determined from the other sample).

As the resin cures, the shrinkage lowers the level of the oil in the pipet. When the level becomes constant, cure is considered complete and the oil level is recorded.

2.2.2 Hardness After Cure

Theory. The post-cure hardness of a resin gives qualitative indication of the ability of a resin to survive thermal and mechanical shock since a semiresilient resin can absorb some of these shock stresses better than a hard, rigid resin. The hardness measurement also gives a qualitative indication of the degree of resin cure since partially cured compounds will be softer than fully cured compounds.

<u>Equipment.</u> This apparatus is the Shore (D) Durometer.

<u>Procedure.</u> Flat-sided samples of the cured resins are used. Hardness readings are taken with the Durometer, with the load applied for 5 sec. The average of five readings is recorded.

2.2.3 Insulation Resistance During Cure

<u>Theory.</u> The insulation resistance of a resin increases during polymerization and reaches a maximum resistance value for the particular resin system. The initial insulation resistance decreases as the temperature of the reactants increases. The resistivity reaches a low point as the exothermic temperature reaches a maximum; as the reaction continues and the exotherm diminishes, the insulation resistance rises rapidly. The curve of insulation resistance vs time is useful for process control and for establishing optimum curing cycles.

<u>Equipment.</u> The three items of equipment needed for this evaluation are:

(1) Test Electrode
The Bell No. 300 Square Test Pattern made from copper-clad, epoxy-glass laminate, specification LAC 22-400., Type 1, Class B, is embedded in the particular resin under test. Insulated conductors are soldered to the contact strips. The Bell No. 300 pattern** is embedded in the resin under test with the copper test pattern functioning as embedded electrodes.

(2) Megohmmeter
A General Radio Co. Megohmmeter Type 1862B is used to determine the insulation resistance of the resin being tested.

(3) Ovens
Mechanical convection ovens are used to cure the resins. The mechanical convection permits rapid heating of the cold molds and aids in dissipating any exotherm developed.

**See photograph in Appendix B.

Procedure. The Bell No. 300 Square Test Pattern is cleaned and placed horizontally in a 3-in. by 3-in. by 0.5-in. silicone mold. The resin is mixed, degassed, and poured into the silicone mold. The filled mold is placed in the oven at the required cure temperature and the leads from the Bell Pattern are connected to the Megohmmeter. A thermocouple is placed in the resin and the temperature is recorded on a recording potentiometer. The insulation resistance is read and recorded every 15 min during the cure. When the insulation resistance reaches a maximum value, the cure is considered complete.

2.2.4 Exotherm

Theory. The exotherm of the resin system is of interest especially when encapsulation of heat-sensitive electronic devices is under consideration. Many so-called room temperature cure materials have exotherms well over 200° F. The exotherm of the resin used to encapsulate an actual module depends on a number of factors such as the following:

(1) Amount of resin
(2) Heat capacity of module components
(3) Thermal conductivity and specific heat of the resin and of the mold
(4) Ambient conditions (i.e., temperature and rate of air flow around the mold)

Equipment. A Varian Recording Potentiometer, with an ice-bath reference junction, is used to record the output of a thermocouple located in the center of the resin mass. The temperature is recorded during the entire cure cycle.

Procedure. A thermocouple is connected to the recording potentiometer and placed in the center of the mold. The resin is mixed, degassed, and poured into the mold. Zero time is taken when the resin is mixed.

In this investigation, samples weighing 50 and 200 gm were run in paper cups; other samples were run in the silicone molds that were used to encapsulate the PAM amplifier assemblies. The samples were cured at temperatures from 80° To 150° F. Some tests were run with the resin preheated to the cure temperature before mixing. The temperature was monitored from zero time through the entire cycle.

2.2.5 Observation Under Polarized Light

Theory. Photo-elastic stress can be observed with a Polariscope* when clear resins are used. The number of stress lines and the spacing between adjacent stress lines indicate stress concentration.

Equipment. The Polariscope consisted of a light source and two layers of polarizing film. The samples were placed between the layers and the lines of stress were visually observed.

Procedure. A Polariscope is used for observation of clear, insulation-resistance samples. The number of isoclinic stress lines is recorded, giving a qualitative indication of the degree of stress.

2.3 ENCAPSULATION

2.3.1 Assembly Processing

Molds were made of silicone rubber for the encapsulation. The assemblies were cleaned in Chlorothene** and the resistance between pins was checked. Two units were dipped in their respective freeze-coats and then cured. The total group of five assemblies was then placed in the molds, the resin was added, and the unit was cured. After

*Photograph in Appendix B
**Chlorothene is a registered trade name, Dow Chemical Corporation.

the resin cured, the resistance between module pins was recorded and resin-hardness readings taken (see Table 2-4 for encapsulation details).

2.3.2 Exotherm

Preheating and exotherm. As indicated earlier, good encapsulation procedure for high-viscosity resins, such as epoxy and polyurethanes, requires that the mixed resin should be preheated, deaerated (degassed), and then poured around the preheated module in its mold. The higher temperatures lower the viscosity of the resin so that it flows easily around the components. The lower viscosity of the preheated resin permits easier deaeration which eliminates voids in the cured module. However, when room-temperature curing agents are used, there is always the danger that the preheating may accelerate the reaction to such a degree that a runaway exotherm may develop and cause damage to heat-sensitive components.

Epocast 202/9615 (110 phr). Epocast 202/9615 has so high a viscosity that it may not be poured cold and must be preheated in order to obtain void-free encapsulations. When it became apparent that Epocast 202/9615 had an exotherm that could cause damage, additional tests were run to minimize the exotherm. The modules were instrumented with thermocuples to determine the actual exotherm in the module where part of the heat is absorbed by the heat capacity of the components and the amount of resin is less than that used for exotherm tests.

Exotherm tests on Epocast 202/9615 (110 phr). Table 2-5 summarizes the series of tests used to explore encapsulation exotherms when this resin was used.

Table 2-4
ENCAPSULATION PROCEDURE

Module No. 103	Module No. 104	Module No. 109	Module No. 108	Module No. 08
Resistance Checked B-A 13K B-D 21K D-B infinity C-A 240K **Cleaned in Chlorothene** **Dipped in Dennis 1162** Cure – 30 min RT 2 hr 150°F 22 hr RT **Encapsulated in 202/9615 cured RT 24 hr** **Resistance Checked** B-A 13K B-D 22K D-B infinity C-A 240K **Shore (D) Hardness 45**	**Resistance Checked** B-A 14K B-D 25.5K D-B infinity C-A 240K **Cleaned in Chlorothene** **Encapsulated in 202/9615 cured 150°F 4 hr** **Resistance Checked** B-A 14K B-D 25K D-B infinity C-A 240K **Shore (D) Hardness 52 (av. of 5)**	**Resistance Checked** B-A 13K B-D 21K D-B infinity C-A 250K **Cleaned in Chlorothene** **Dipped in Dow Corning 271 cured 24 hr RT** **Encapsulated in Stycast 2651 cured 2 hr at 165°F** **Resistance Checked** B-A 13K B-D 22K D-B infinity C-A 240K **Shore (D) Hardness 88**	**Resistance Checked** B-A 14K B-D 21K D-B infinity C-A 250K **Cleaned in Chlorothene** **Encapsulated in Stycast 2651 cured 2 hr at 165°F** **Resistance Checked** B-A 14K B-D 22K D-B infinity C-A 240K **Shore (D) Hardness 88**	**Resistance Checked** B-A 12K B-D 21K D-B infinity C-A 250K **Cleaned in Chlorothene** **Encapsulated in PR 1538 cured 16 hr at 150°F** **Surface uncured – sticky post-cured 48 hr at 150°F** **Surface still uncured – removed mechanically** **Re-encapsulated cured 16 hr at 150°F post-cured 48 hr at 150°F** **Surface uncured – tacky, no adhesion to old material** **Resistance checked** B-A 11K B-D 20K D-B infinity C-A 230K **PR 1538 stripped in Methyl-ethyl Ketone (two resistors and large glass capacitors broken during stripping; they were replaced)** **Resistance checked** B-A 13K B-D 20K D-B infinity C-A 230K **Re-encapsulated in PR 1538 cured 150°F for 16 hr** **Resistance checked** B-A 16K B-D 24K D-B infinity C-A 220K **Shore (A) Hardness 77**

Table 2-5

EXOTHERM TESTS ON EPOCAST 202/9615

Amount of Resin (gm)	Mold Material	Resin Mix Preheated (°F)	Curing Temp. (°F)	Peak Exotherm (°F)	Figure No.
200 gm	Paper cup	Yes at 150°F	150°F oven	345	3-4
200 gm	Paper cup	No	150°F oven	314	3-5
200 gm	Paper cup	No	77°F	176	3-6
50 gm	Paper cup	Yes at 150°F	150°F oven	310	3-4
50 gm	Paper cup	No	150°F oven	275	3-5
50 gm	Paper cup	No	77°F	93	3-6
60 gm	Silastic Mold	Yes at 150°F	150°F oven	290	3-12
60 gm	Silastic Mold	No	77°F	85	3-6
Module No. 104	Silastic Mold	Yes at 150°F	150°F oven	252	3-7
Module No. 103	Silastic Mold	Yes at 150°F	77°F	262	3-7

As can be seen from Table 2-5, the heating required to successfully encapsulate a module was sufficient to initiate the exothermic reaction. Because of the increased air flow, and consequently the better heat-transfer rate, the module that was cured in a mechanical convection oven had an exotherm peak that was 10°F less than the module that was cured on a bench top at room temperature.

2.3.3 Cure Inhibition of PR 1538

Surface-cure inhibition was encountered with PR 1538. This inhibition was caused by the silicone rubber mold absorbing the curing agent from the polyurethane resin.

The uncured surface material was mechanically removed and the module recast. Surface-cure inhibition was again encountered in the second encapsulation and the new material did not bond well to the old; it was necessary to strip the module of all the encapsulant and to reencapsulate in a mold that had been saturated with the PR 1538 curing agent. A capacitor and a resistor were damaged during the stripping operation. These items were replaced, resistance measurements taken, and the module reencapsulated in PR 1538.

Section 3-1
EXPERIMENTAL RESULTS

The data presented in this section, while referring directly to the testing of the resins only, has its obvious application to resins as they are used to encapsulate the PAM amplifier modules.

3.1 SHRINKAGE DURING CURE

Table 3-1 presents data that results from use of the technique described in Section 2.2.1.

Table 3-1

SHRINKAGE DURING CURE

	2651 MM	PR 1538	202/9615
Reading at Gel Point (ml)	0.58	0.45	0.17
Reading at Cure (ml)	0.97	0.60	1.10
Change in Volume (ml)	0.39	0.15	0.93
Density of Resin System (150° F) at Maximum Volume	1.528	1.054	1.018
Volume of 25 gm Resin (ml)	16.4	23.7	24.3
Percent Volume Shrinkage During Cure (after gelation) (%)	2.38	0.63	3.78

3.2 HARDNESS AFTER CURE

Section 2.2.2 gives a description of equipment and test procedures used.

Table 3-2

HARDNESS AFTER CURE

Resin	Shore D
2651 MM	88
PR 1538	24
202/9615	49

3.3 INSULATION RESISTANCE DURING CURE

These data for Stycast 2651 MM, Epocast 202/9615 (110 phr), and PR 1538 are described in Figs. 3-1, 3-2, and 3-3, respectively, using coordinates of Temperature vs Time vs Resistance.

3.4 EXOTHERM

The curves in the figures displayed in this section are the experimental results of procedures discussed in Sections 2.2.3 and 2.2.4.

3.4.1 Exotherm-Epocast 202/9615 (110 phr)

(1) Material preheated to 150° F and cured at 150° F, Fig. 3-4.
(2) Material poured at 77° F and cured at 150° F, Fig. 3-5.
(3) Material poured at 77° F and cured at 77° F, Fig. 3-6.
(4) Exotherm curves on modules No. 103 and No. 104, Fig. 3-7.

3.4.2 Exotherm-Stycast 2651 MM

(1) Material preheated to 150° F and cured at 150° F, Fig. 3-8.
(2) Material poured at 77° F and cured at 150° F, Fig. 3-9.

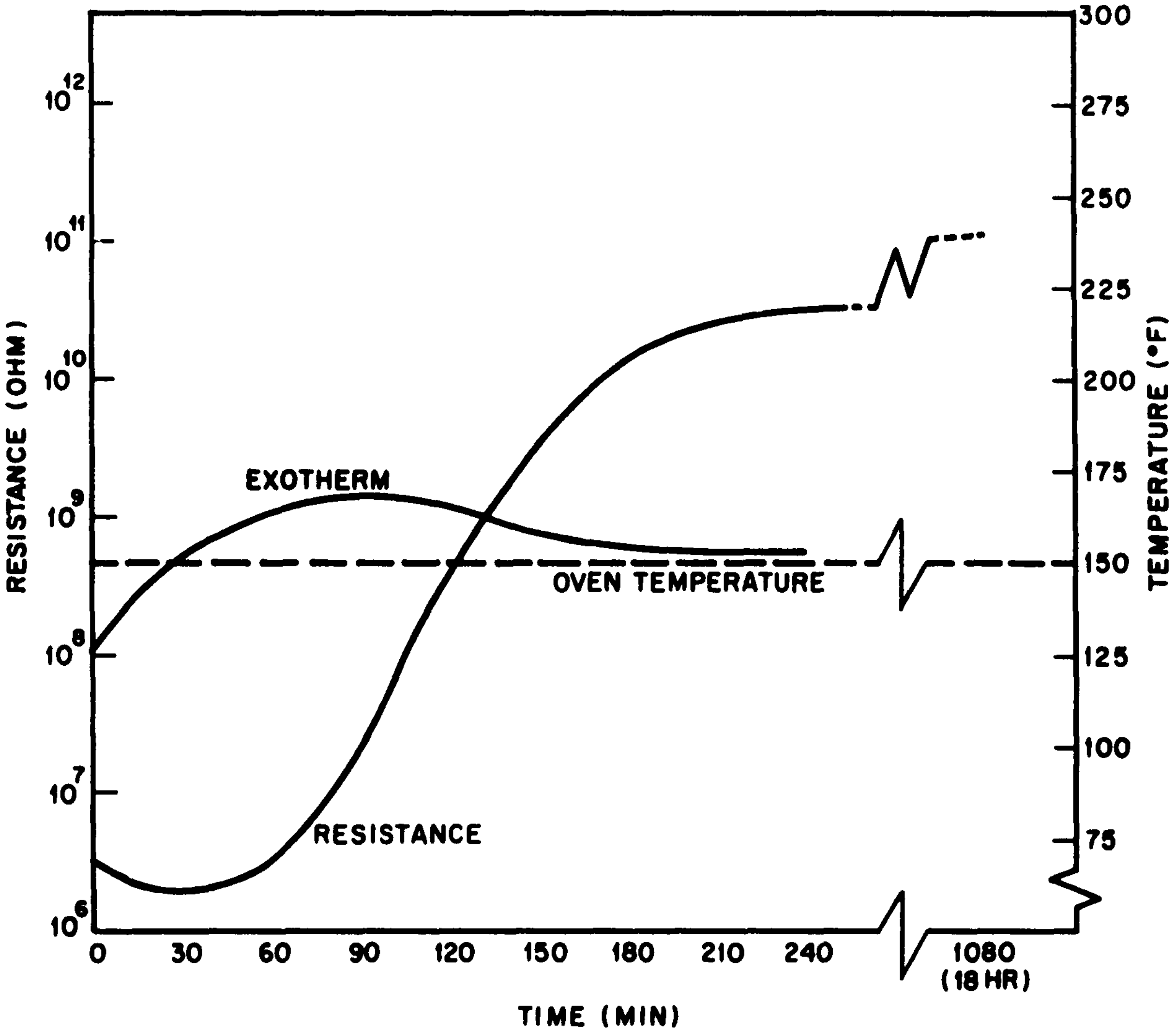

Fig. 3-1 Insulation Resistance, Exotherm of Stycast 2651 MM

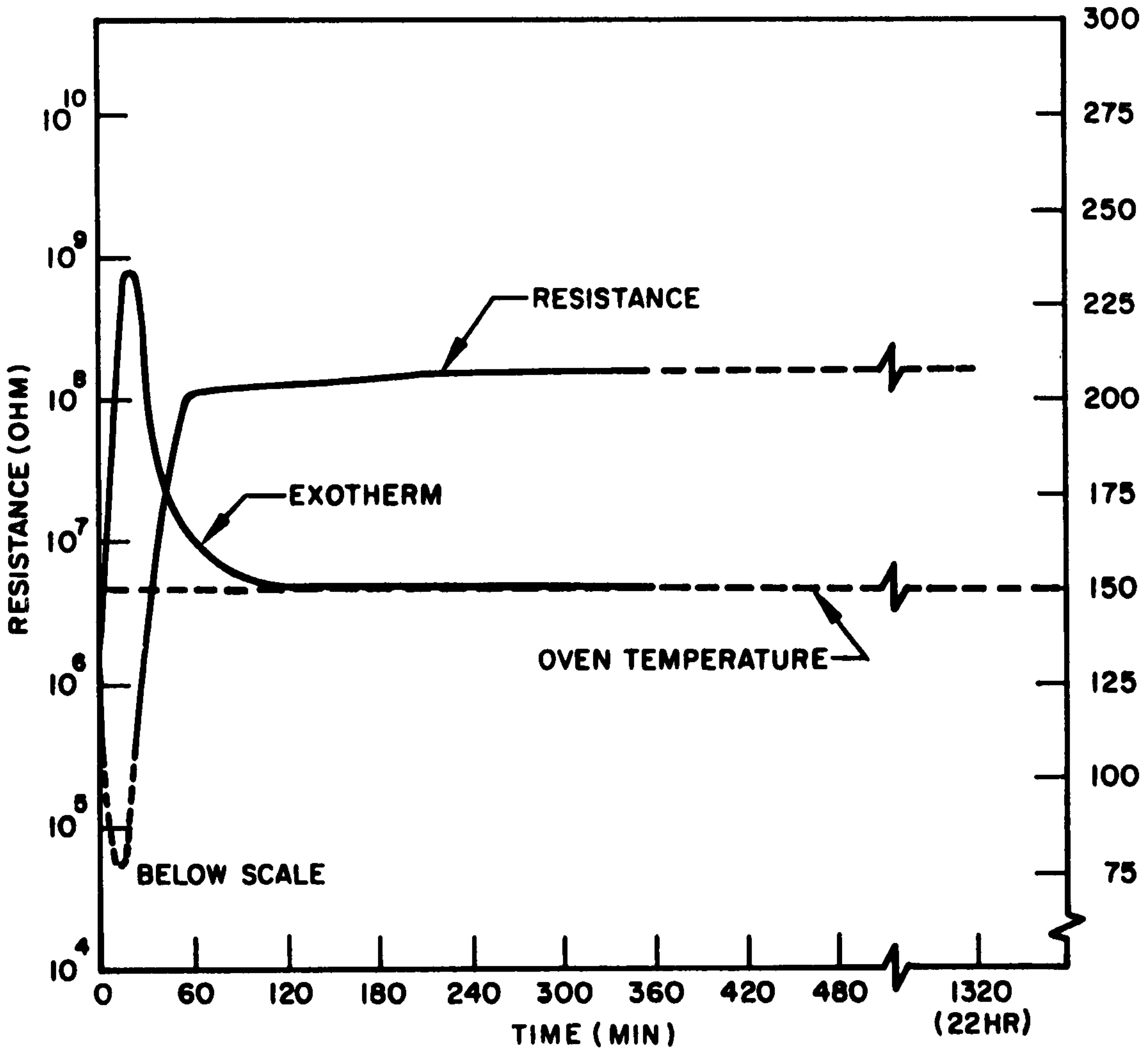

Fig. 3-2 Insulation Resistance and Exotherm Curve of Epocast 202/9615

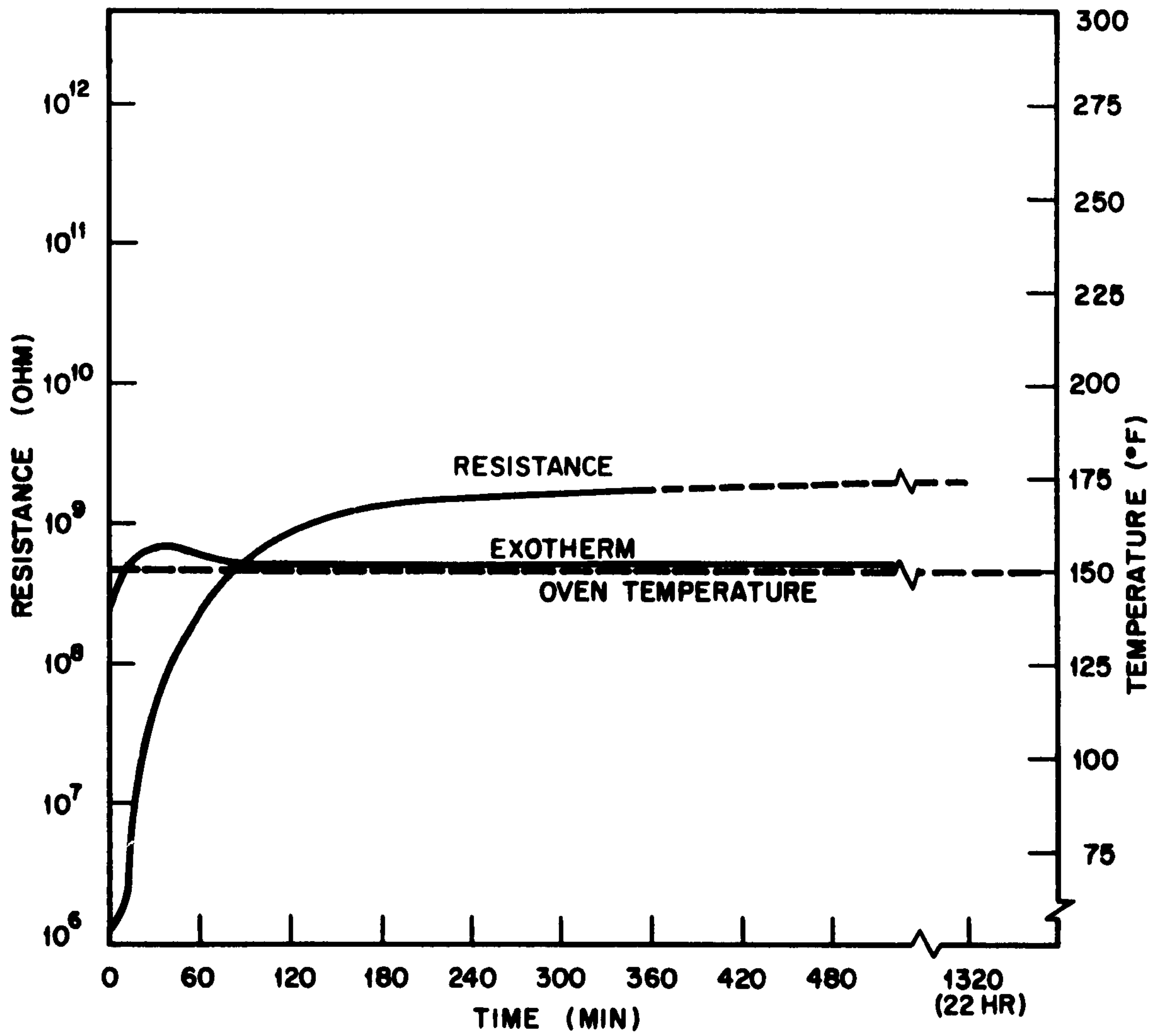

Fig. 3-3 Insulation Resistance and Exotherm Curve on PR 1538

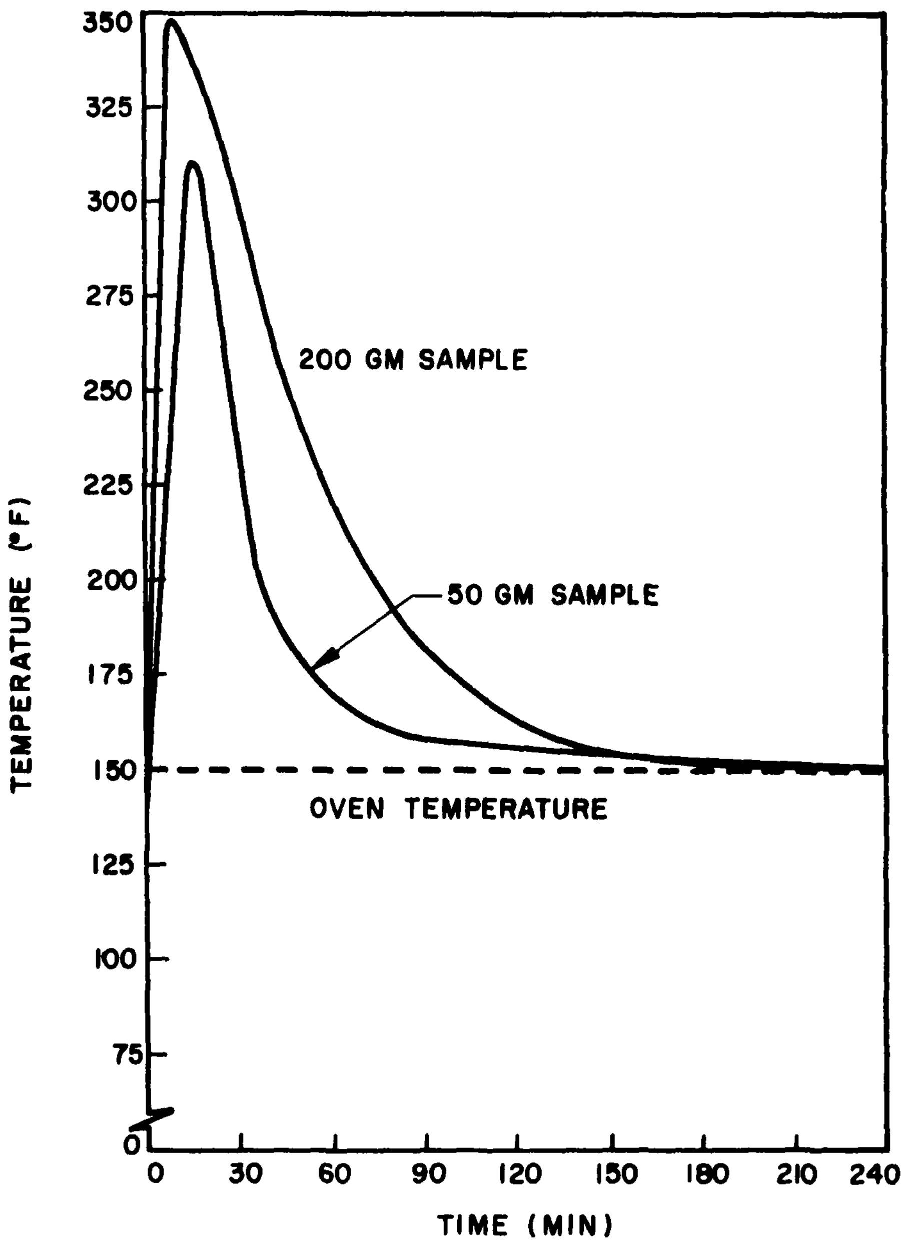

Fig. 3-4 Exotherm Curve of 202/9615 at 150° F in a Paper Cup (Material Preheated to 150° F Before Mixing)

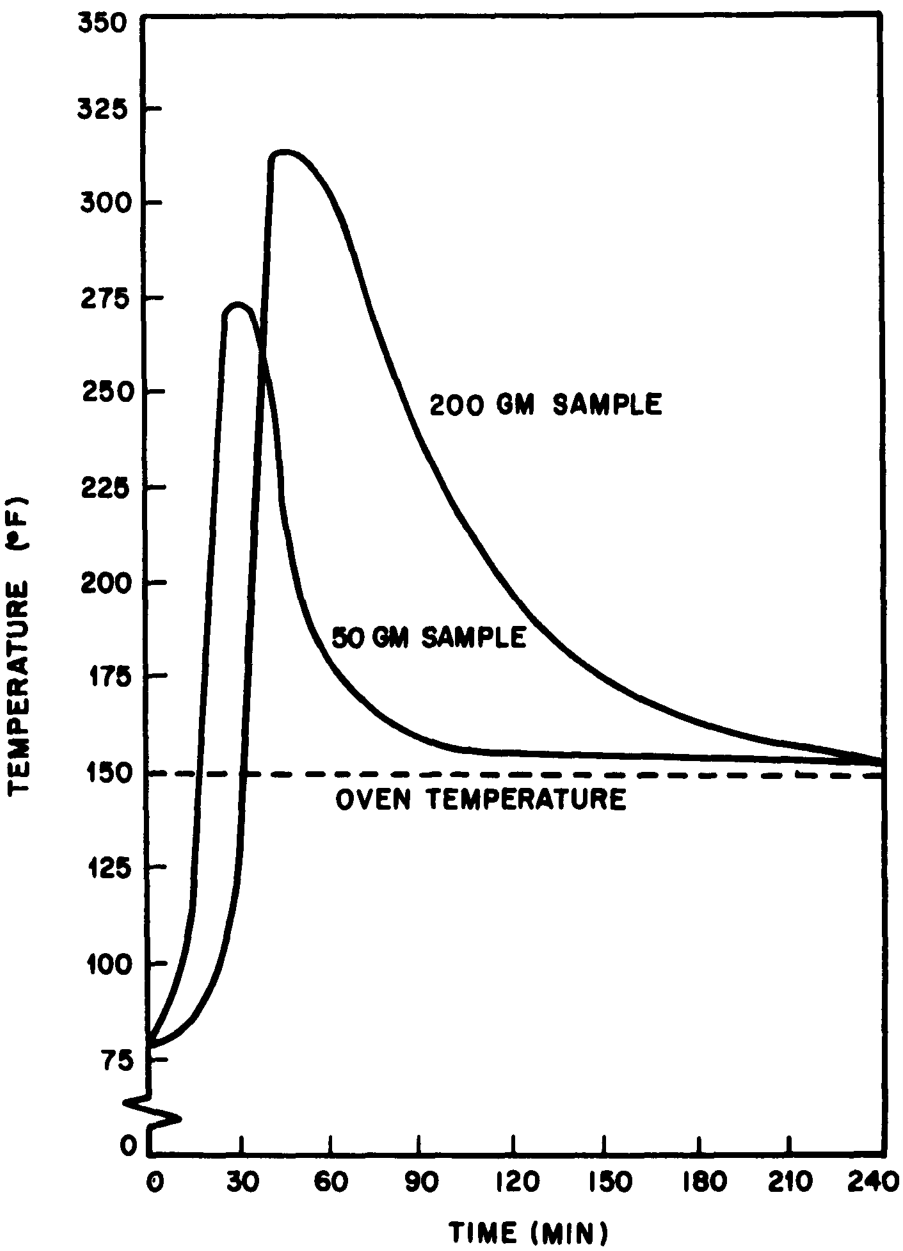

Fig. 3-5 Exotherm Curve of 202/9615 at 150° F in a Paper Cup (Material Mixed at Room Temperature)

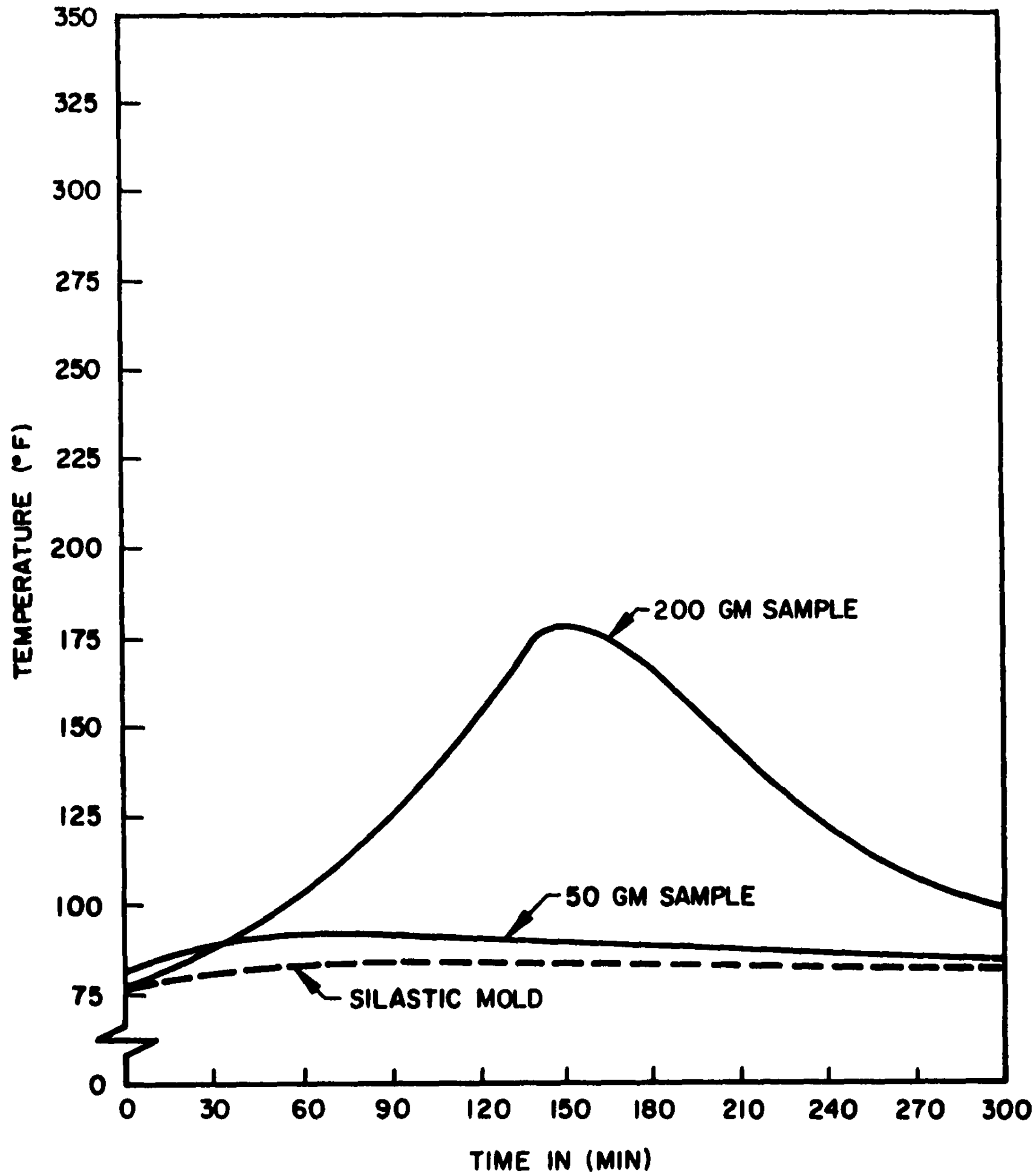

Fig. 3-6 Exotherm Curves of 202/9615 With Room Temperature Cure (Material Mixed at Room Temperature)

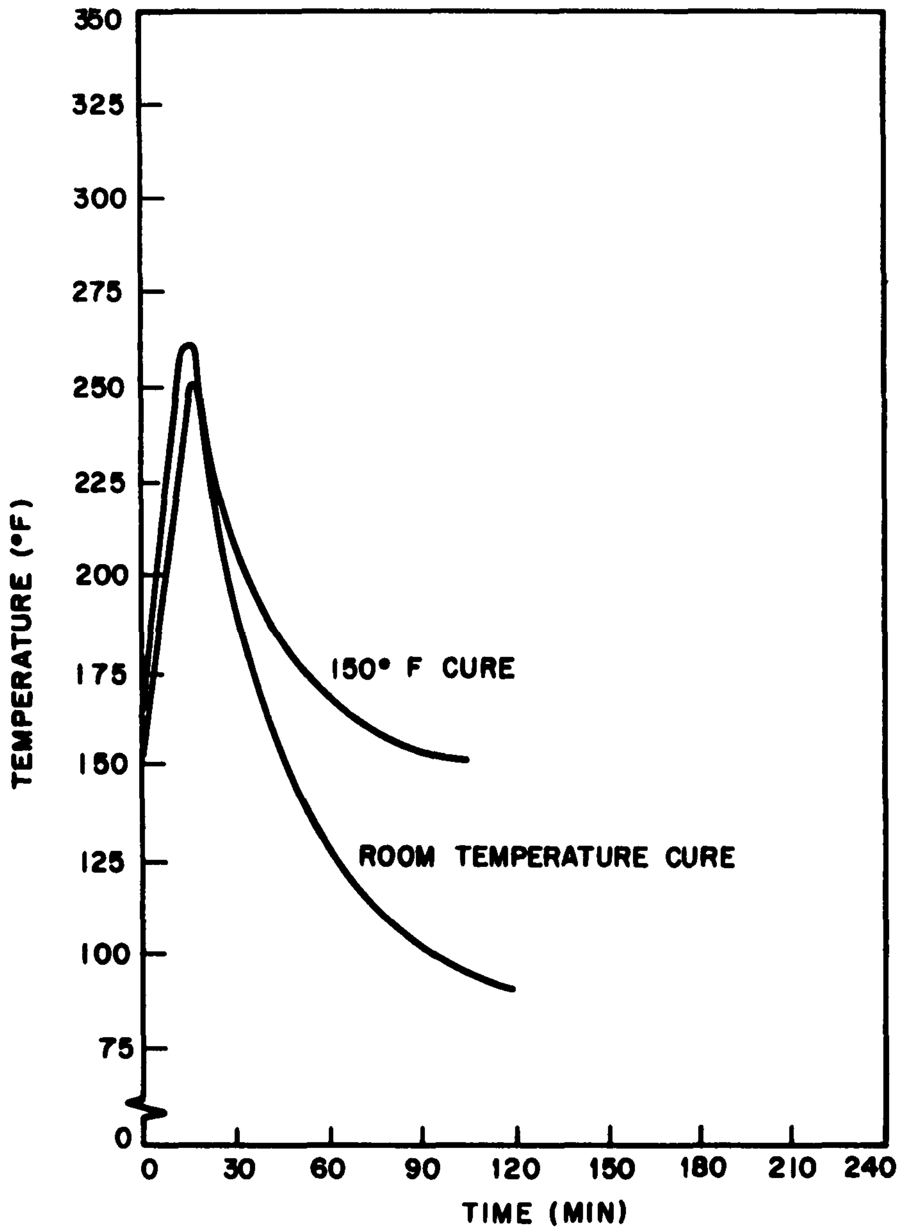

Fig. 3-7 Exotherm Curve of 202/9615,Silastic Mold With Module (Material Preheated to 150° F)

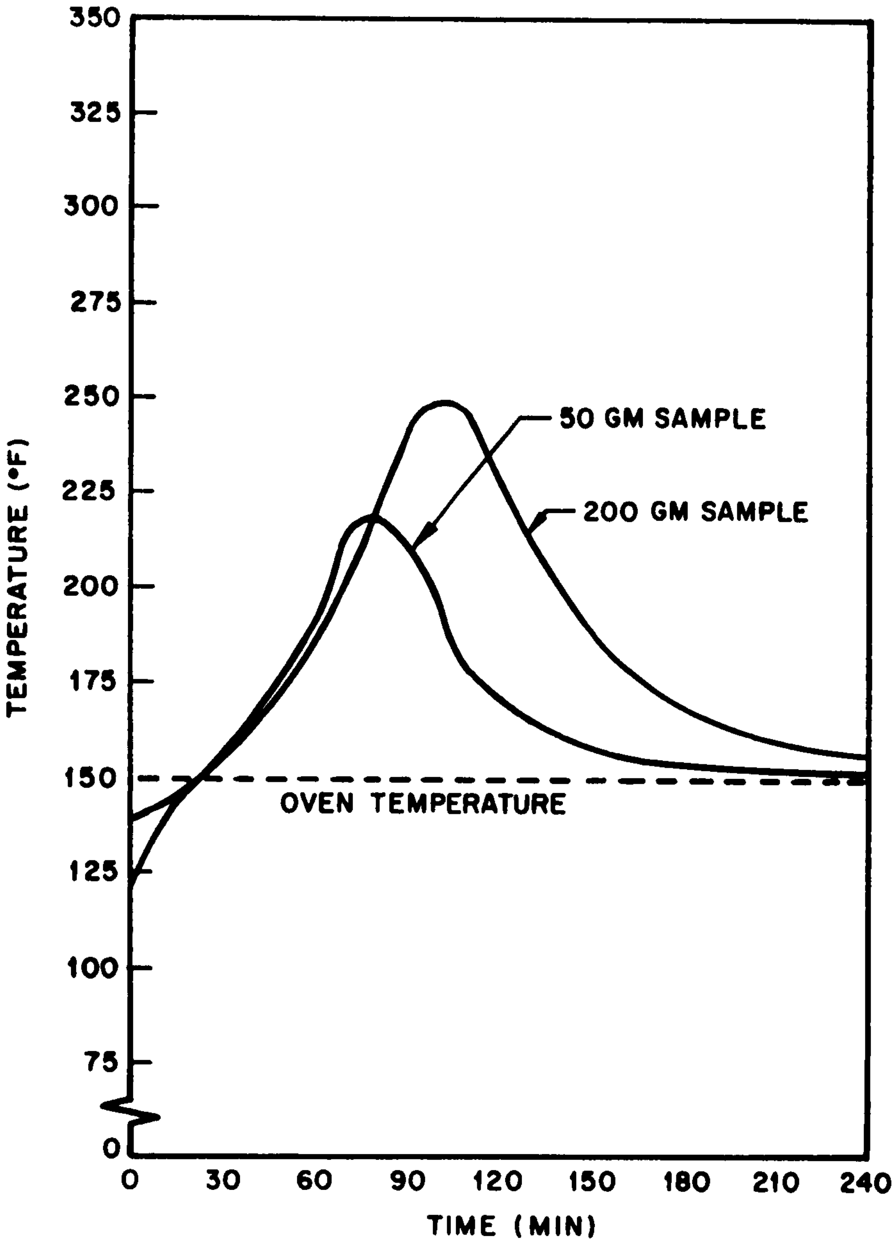

Fig. 3-8 Exotherm Curve of Stycast 2651 MM at 150° F in a Paper Cup (Material Heated to 150° F Before Mixing)

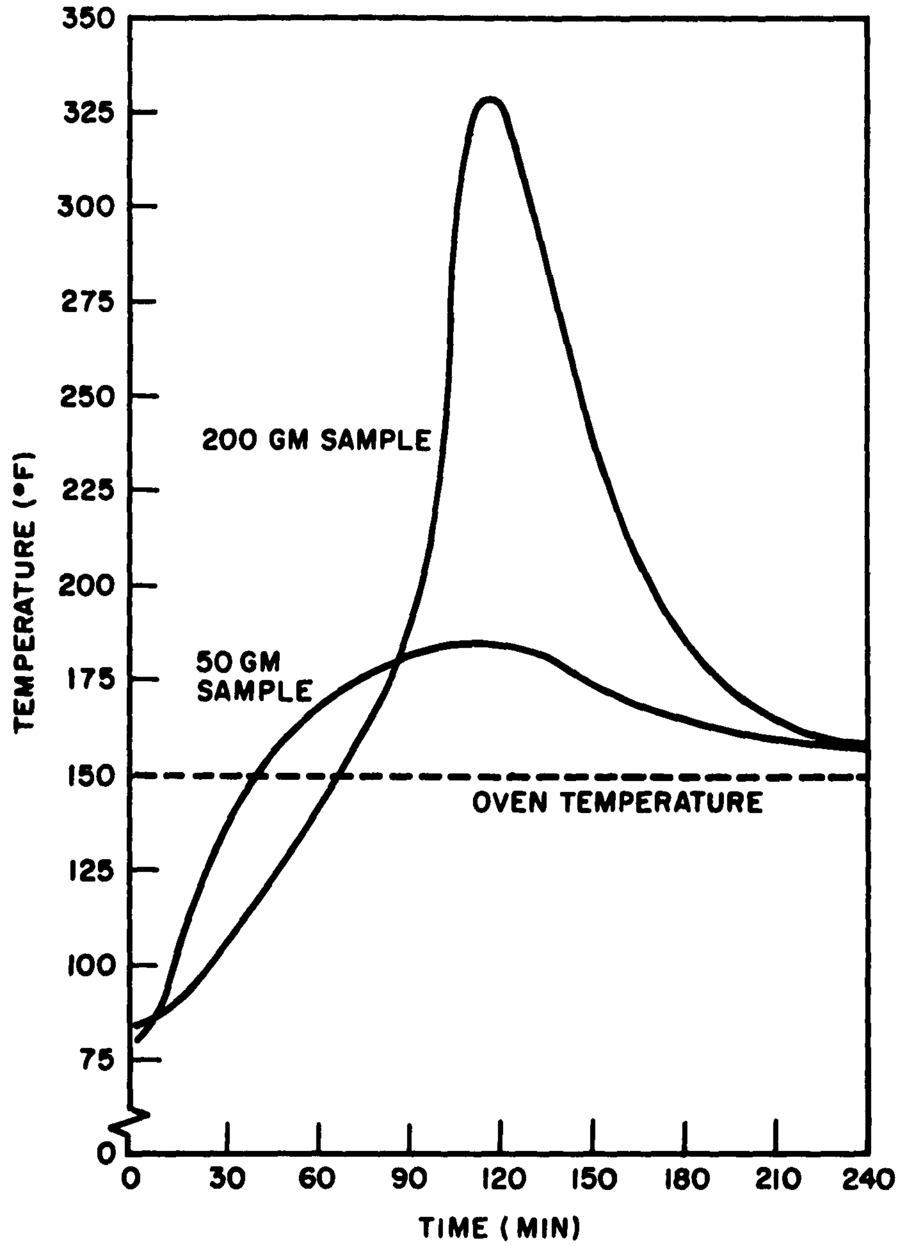

Fig. 3-9 Exotherm Curve of Stycast 2651 MM at 150° F in a Paper Cup (Material Mixed at Room Temperature)

3.4.3 Exotherm-PR 1538

(1) Material preheated to 150°F and cured at 150°F, Fig. 3-10.
(2) Material poured at 77°F and cured at 150°F, Fig. 3-11.

3.4.4 Exotherm-Silastic Mold

The three resins were preheated to 150°F, cast into a preheated (150°F) silastic mold and cured in an oven at 150°F. Figure 3-12 displays the results.

3.5 POLARIZATION

Stress lines observed around the encapsulated insulation-resistance epoxy board are enumerated in the following table.

Table 3-3

STRESS LINES IN RESINS AFTER CURE

Material	Cure Schedule	Post Cure	No. of Stress Lines
Stycast 2651 MM	18 hr/150°F	None	Opaque
PR1538	22 hr/150°F	None	0
Epocast 202/9615	22 hr/150°F	None	4
Epocast 202/9615[a]	72 hr/77°F	None	0
Epocast 202/9615[a]	72 hr/77°F	2 hr/185°F	1

(a) Same sample

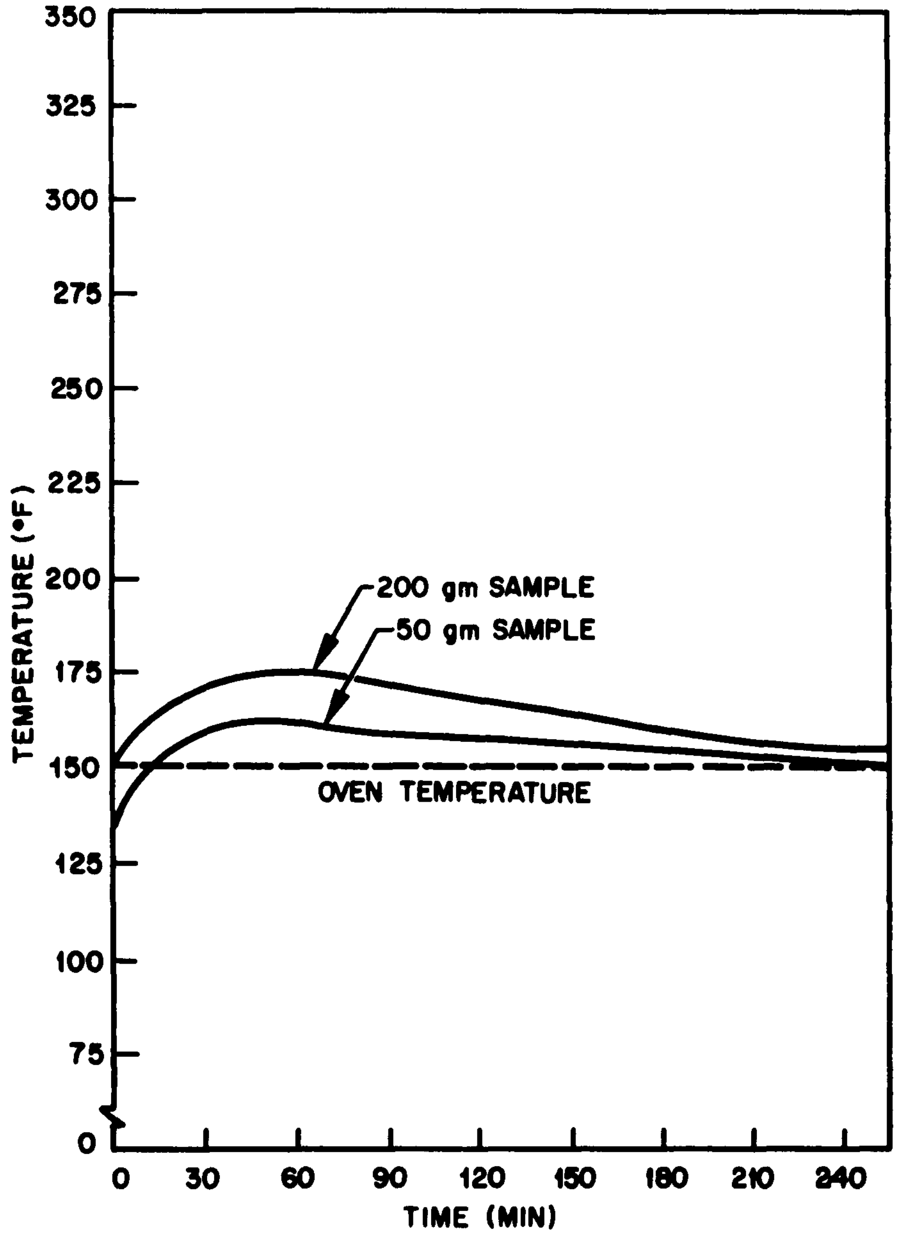

Fig. 3-10 Exotherm Curve of PR 1538 at 150° F in a Paper Cup (Material Preheated to 150° F Before Mixing)

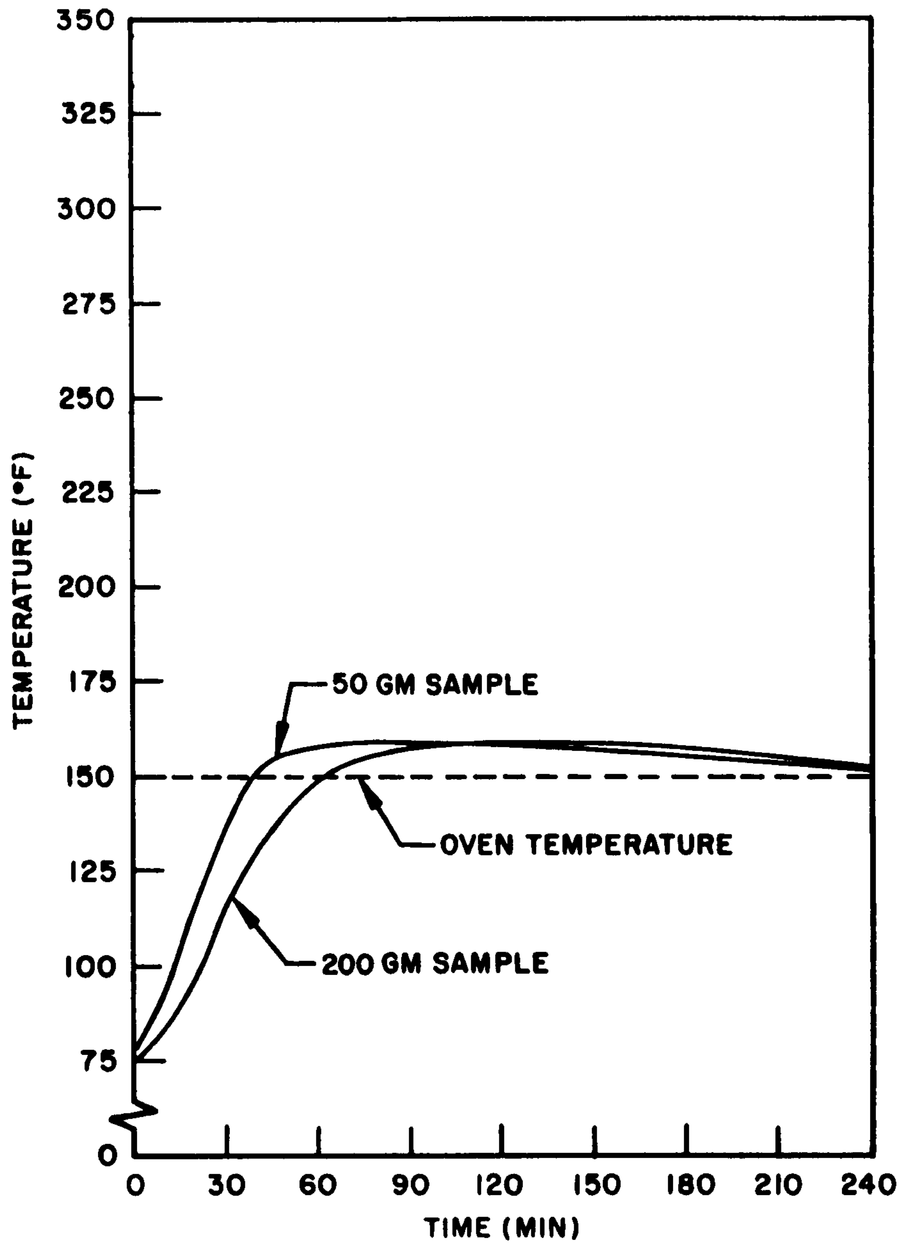

Fig. 3-11 Exotherm Curve of PR 1538 at 150° F in Paper Cup (Material Mixed at Room Temperature)

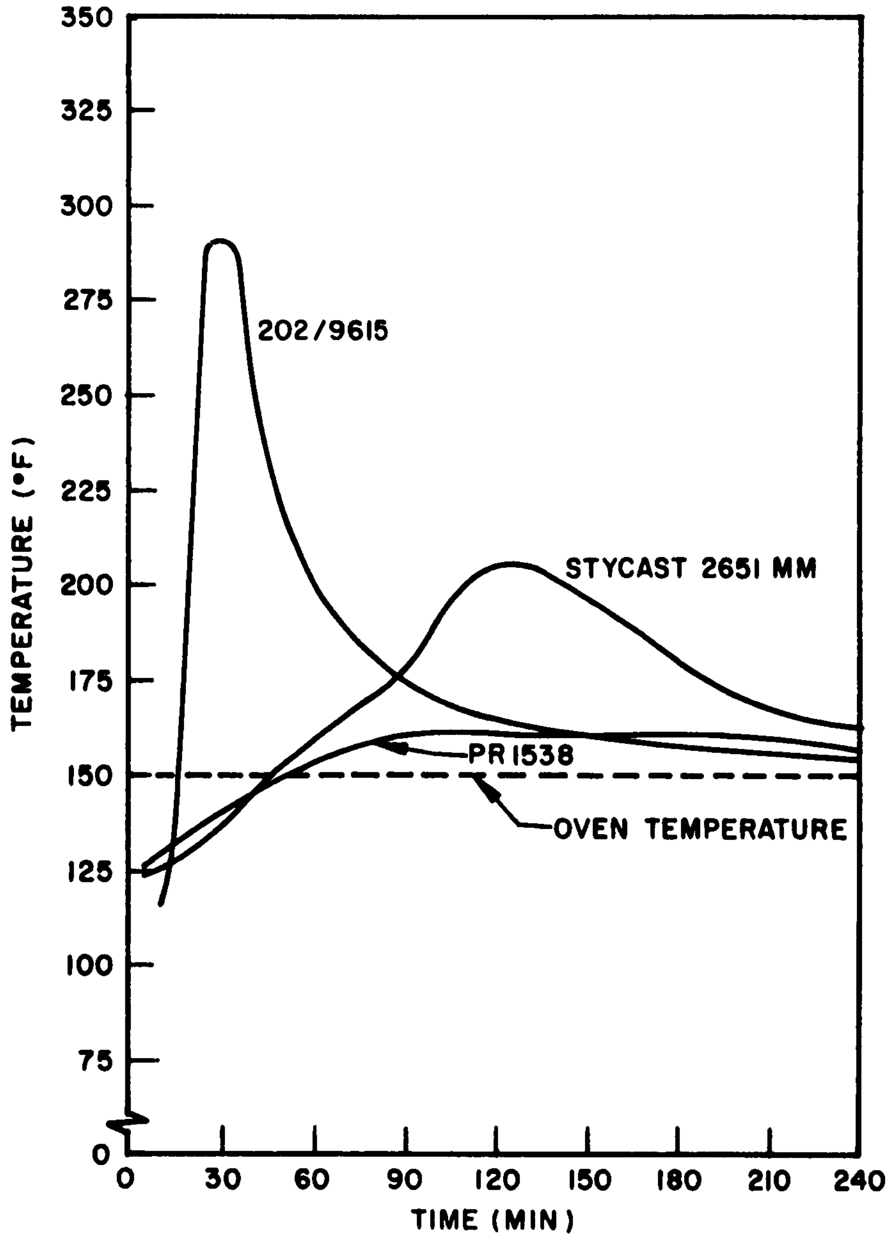

Fig. 3-12 Exotherm Curves of Samples in Silastic Mold at 150° F

Section 4
DISCUSSION

4.1 INSULATION RESISTANCE

The embedded Bell No. 300 Square Test Pattern is currently being used by Space Systems* as a Quality Assurance test. The insulation-resistance test serves as a check on the following:

(1) The quality of the resin system
(2) The accuracy of the proportioning operation (weighing out of resin and catalyst)
(3) The efficiency of the mixing or blending operation

A bad batch of resin or curing agent (contaminated with ionic contaminants or with moisture) can easily be detected by its insulation resistance under a given set of cure conditions. A resin system that is improperly proportioned, poorly mixed, or incompletely cured will generally have a lower than normal insulation resistance.

The data shown in Figs. 3-1, 3-2, and 3-3 demonstrate that the insulation test with the embedded Bell No. 300 Square Test Pattern can be used to establish optimum curing cycles. The present laboratory technique (e.g., connecting the electrode wires directly to a megohmmeter and taking readings every 15 min) is tedious and time consuming especially when longer curing-cycle resins are used. The technique is not easily automated since the test voltage is applied for only a one minute interval every 15 min and the range of resistance varies from 5×10^5 ohm to 2×10^{12} ohm or greater. Once the insulation-resistance curve has been plotted for a particular curing cycle, it is a simple matter to vary the curing cycle, measure the insulation resistance at certain points, and compare these to the original curve.

*Lockheed Aircraft Corporation Process Specification No. LAC 0419A, "Plastics For Application to Electrical and Electronic Components and Assemblies."

4.2 EXOTHERM

As was pointed out in Section 2.2.4 of this report, the actual exotherm in a module depends on a number of factors such as amount of resin, heat capacity of module components, thermal conductivity and specific heat of the resin and the mold, and ambient curing conditions. Exotherm can be reduced, with a particular module and encapsulant in use, by following this method:

(1) Keep the amount of resin to a minimum.
(2) Use high thermal conductivity molds (i.e., metal).
(3) Increase air flow around the mold (e.g., use mechanical convection ovens).
(4) Gel the resin at a lower temperature and then post cure for final properties.

From the standpoint of money and time expenditure, especially in shortrun production or prototype work, it is easier to justify the choice of a resin with a lower exotherm rather than the choice of fabrication of expensive female metal molds or programming curing cycles.

4.3 SHRINKAGE DURING CURE

The polyurethane, PR 1538, had the lowest volume shrinkage (0.63 percent) of the three resin systems. The manufacturer's data sheet (Table 2-3) claims a volume shrinkage of 1.4 percent. The manufacturer arrives at this figure by inserting the room-temperature specific gravities of liquid-catalyzed resin and solid-cured resin into the following equation:

$$1 - \frac{\text{Density uncured}}{\text{Density cured}} \times 100 = \text{percent volume shrinkage}$$

This technique gives total shrinkage and does not separate the shrinkage of the resin in the liquid state from the shrinkage after gelation.

The dilatometer technique is very sensitive to temperature variations due to the large coefficient of expansion of the liquid resin and of the silicone oil. Protection from convection heat losses is afforded by close control of the constant-temperature bath and by a shield for the protruding pipette dilatometer.

4.4 POLARIZATION

The resilient polyurethane resin, PR 1538, cured around the insulation-resistance board without developing stress lines. The Epocast 202/9615 sample, cured at room temperature, did not develop stress lines until it was post cured for 2 hr at 185°F. The increased number of stress lines is indicative of an incomplete cure at room temperature. The same material cured at 150°F had four stress lines, the increased stress being due to the greater degree of cure at 150°F plus the thermal shrinkage of the cured resin from curing temperature to room temperature.

Section 5
CONCLUSIONS

5.1 PR 1538

This material cures to a low shrinkage, strain-free, resilient, tough, and abrasive-resistant polyurethane elastomer. It has poor repairability due to the toughness of the material; this toughness makes it extremely resistant to carving or abrasion and reduces its adhesive qualities. The high coefficient of thermal expansion, 24×10^{-5} in/in/°C, may present a problem in the Paraplate type of electronic packaging (e.g., components stacked between two printed, wiring boards).

The adhesive qualities between PR 1538 and a number of substrates is poor, necessitating the use of a primer where a good adhesive bond is required. However, when the PAM amplifiers were encapsulated, the resin totally encapsulated all components and no primer was used. In addition, the poor bond of this material has the advantage that there is less tendency for the resin to pull components apart during thermal cycling.

The surface inibition encountered when the silicone rubber molds were used presented a problem in prototype work where this type of mold was used almost exclusively. In production, where metal molds or epoxy molds are used, surface inhibition, would not be a problem.

5.2 STYCAST 2651 MM

This material is a hard, rigid, semifilled, low viscosity, opaque, epoxy resin. Because of its low viscosity and small amount of filler, Stycast 2651 MM is easily used for potting or encapsulating. Isoclinic stress lines cannot be observed because of the opacity of the resin.

5.3 EPOCAST 202/9615 (110 phr)

High shrinkage, high viscosity, high exotherm, and medium stress are characteristics of this material when it is cured at 150° F. It is possible to cure this resin at room temperature and obtain a strainfree, embedded, insulation-resistance board sample; however, its high viscosity precludes its use as an electronic-module encapsulant without heating (150° F). Heating at this temperature is usually sufficient to initiate the undesirable exothermic reaction and to shorten the usable pot life to approximately 10 min (105 gm mass).

It is possible, under certain conditions, to use this resin system without generating an excessive exotherm. For example, it is possible to preheat resin and curing agent to 150° F, weigh out (105 gm total), mix, degas, and pour into a silastic insulation resistance mold and cure at room temperature on a bench top without generating any exotherm whatever. This particular mold has a large surface area (9 in.^{2}) and is only 0.5 in. thick. The exothermic heat is rapidly dissipated from the large surface area. Similarly, it may be possible, by using a small metal mold, to preheat resin, degas, and pour into a hot mold and cure in an air circulating oven at room temperature without generating an appreciable exotherm.

5.4 FREEZE-COATING MATERIALS

The evaluation of the effectiveness of the two freeze-coating materials can only be done by environmental testing of the encapsulated modules. The Dennis 1162 resin may help to distribute the compressive and tensile stress concentrations during thermal cycling. The Dow Corning 271 Adhesive, in addition to distributing any compressive or tensile stress concentration, functions as a release agent and prevents the resin from bonding to the components. The extent to which these materials function can be determined only by thermally cycling the encapsulated modules.

Section 6
RECOMMENDATIONS

These recommendations are made for Epocast 202/9615 (110 phr):

(1) Exotherm tests should be run on this resin for each specific application where the use of this material is desired.

(2) Insulation-resistance and hardness measurements should be taken to determine if there will be age-hardening effects especially with the room-temperature cured material.

A recommendation is made for an additional Experimental Memo to instigate the investigation of methods for measuring stress induced in an encapsulating material as a result of resin shrinkage and thermal cycling. The Polariscope can display isoclinic stress lines only when transparent materials are being studied.

It is further recommended that all modules be thermally cycled to the failure point rather than to the minimum number of cycles called out in ATP-MO 68118 (see EM 2110, Appendix).

APPENDIX A

LOCKHEED AIRCRAFT CORPORATION-MISSILES and SPACE DIVISION

EXPERIMENTAL MEMO No. 2110

TITLE: INVESTIGATION OF ELECTRONIC MODULE POTTING RESINS				SPARES REQUIRED [ ] YES [ ] NO
FROM (Organization name and number): Value Engineering, 62-81	MODEL NO.: 5205-2204 & up	ORIGINATOR'S NEED DATE: 11/25/61	ESTIMATED COMPLETION DATE: 1/18/61	SCHEDULE APPROVAL: W. Yeggy 10/31/61
ORIGINATOR: C. F. MacLean DATE: 10-24	USAGE: Test	ESTIMATED MAN HOURS: 320	EST. MATL. DOLLARS: $475.00	BUDGET APPROVAL: R. B. Bryant
TO (Organization name and number): Reliability, Dept. 61-26	TYPE OF INSPECTION: N/A	APPROVAL ORIGINATING ORGANIZATION — NAME	DATE	APPROVAL RECEIVING ORGANIZATION — NAME / DATE
ATTENTION OF: A. T. Hamala, Dept. 61-26	CONTACT: D. Kasper	E. C. Hart		
CLASS: 31 W.O. NUMBER: 3821 W.A. NUMBER: 6000		W. J. Sullivan		
DESTINATION OF PARTS OR OTHER ITEMS: Originator	EXT. 2-8788			

DESCRIPTION OF JOB

Scope: To investigate characteristics of several potting compounds when subjected to thermal shock and cycling. Also, the possible effects of the potting compound on the electrical functioning of a selected module will be investigated.

Parts and Material Identification: All parts and materials used in the performance of these tests shall be identified and recorded. Identification and the record shall include the following:

Manufacture
Lot or Unit
Material or Part Name
Used for specification no. ______ thru no. _______.

Parts and Materials:

A. **Parts**
   1. PAM amplifier assy (per Dwg. 1322517)
   2. Same as (1) except a different potting compound as may be specified.

B. Thin Coating (or Freeze Coat)
   1. LAC 37-4070 Class 1
   2. Dow Corning 271 Adhesive

C. Potting Compound
   1. LAC 40-4093
   2. LAC 40-4088
   3. Products Research PR-1538

REMARKS:

cc: C. F. MacLean, 62-81, 104 (5)

FORM LMSD 1264-1

EM No. 2110

Page 2 of 2

Requested Tests (see note below):

1. Tests to be performed on the resin

    a. Shrinkage during cure
    b. Hardness after cure
    c. Insulation resistance during cure, readings to be taken every 10 minutes for the first hour and every 15 minutes thereafter until a constant value is reached. If constant value not reached after 8 hours – take readings several times a day until a constant value is reached.
    d. Exotherm on 50 gram and 200 gram samples, both temperatures to be 150° F.
    e. Observe under polaroid light possible internal stresses with and without compressive loading.

2. Thermal shock – use ATP – M068118
    Check electrical continuity before, during, and after test. Note any cracks or other degradation of the potting compound.

Note: For detail of test methods and source of modules, contact D. Kasper, Ext. 2-8788, Bldg. 104.

Test Report: Final report required.

LOCKHEED AIRCRAFT CORPORATION–MISSILES and SPACE DIVISION

EXPERIMENTAL MEMO No. 2110-1

TITLE INVESTIGATION OF ELECTRONIC MODULE POTTING RESINS				SPARES REQUIRED ☐ YES ☐ NO
FROM (ORGANIZATION NAME AND NUMBER) Value Engineering, 62-81	MODEL No. 5205 2204 & Up	ORIGINATOR'S NEED DATE 12-22-61	ESTIMATED COMPLETION DATE 12-22-1	SCHEDULE APPROVAL
ORIGINATOR C.F. MacLean — DATE 11/17	USAGE Test	ESTIMATED MAN HOURS	EST. MATL. DOLLARS	BUDGET APPROVAL J. Somers
TO (ORGANIZATION NAME AND NUMBER) Reliability, Dept. 61-26	TYPE OF INSPECTION N/A	APPROVAL ORIGINATING ORGANIZATION — NAME	DATE	APPROVAL RECEIVING ORGANIZATION — NAME / DATE
ATTENTION OF X2-8708 A.T. Hamala, 61-26,	CONTACT: D. Kasper	E. C. Hart		D. H. Meylink 11/29/61
CLASS 31 / W.O. NUMBER 3521 / W.A. NUMBER 9705-01				
DESTINATION OF PARTS OR OTHER ITEMS Originator	EXT. 2-8788			

DESCRIPTION OF JOB

This EM is in addition to and to be coordinated with EM-2110, that is worked concurrently. This EM does not supersede any part of EM-2110. The intent of the EM (2110-1) is to clarify and supply the required data to get the desired modules built.

Add the following under: Parts and Materials,) Parts;

Parts Required – PAM Amplifier Assy
Dwg. 1322517 – except no potting compound applied
Qualtity – 5 total
Electrical test to applicable portions of ATP – MO 68118
Need date for this part – 12-22-61.

This EM also substitutes a new WO and WA number for this assignment. 3521-9705 is a common pool charge number assigned to Dept. 62-81, and is not chargeable to any one program. Therefore, only the one authorizing signature of budget approval is required.

REMARKS:

cc: C.F. MacLean, 62-81, 104 (5)

FORM LMSD 1264-1

APPENDIX B

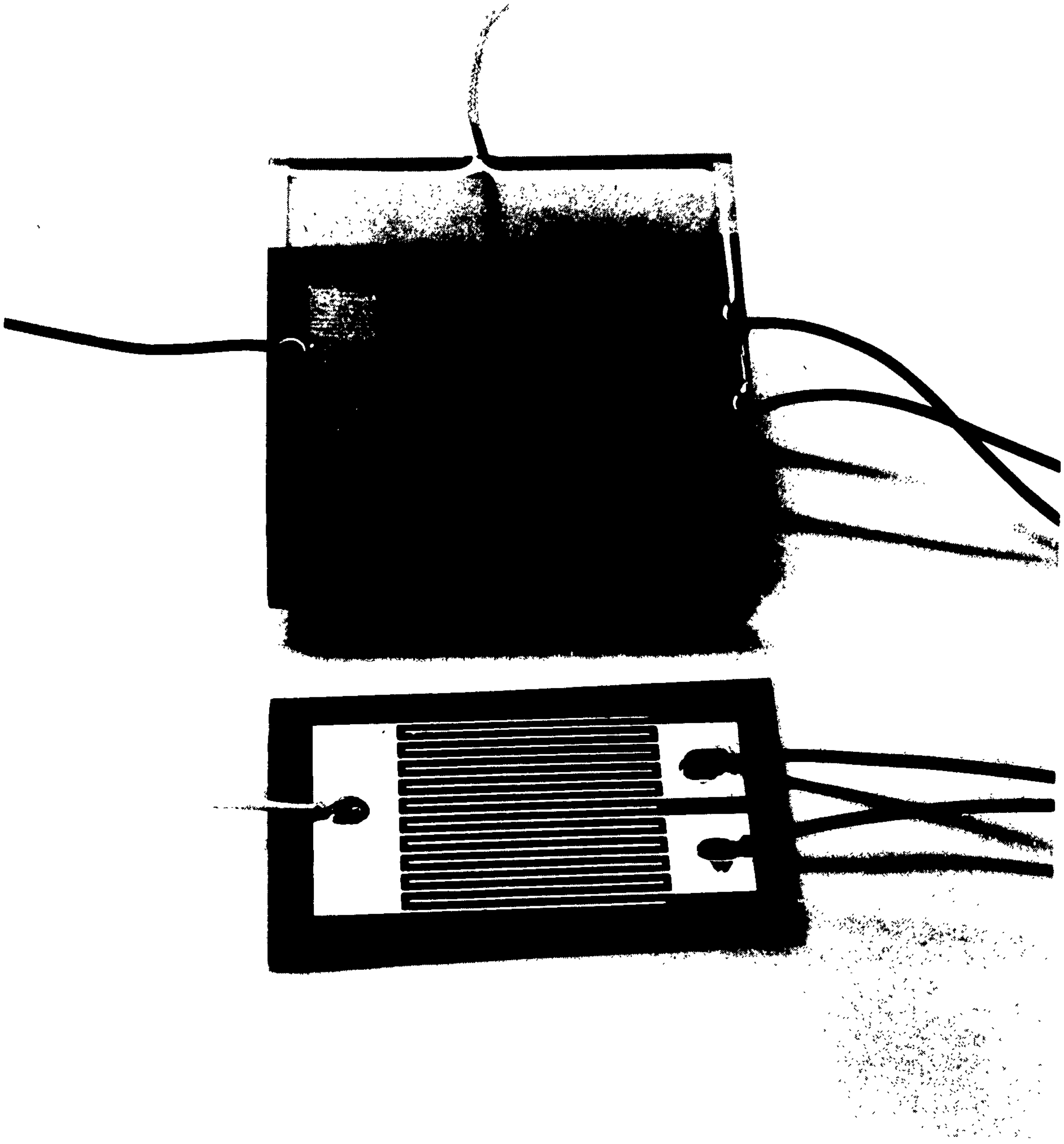

Fig. B-1 Bell No. 300 Pattern Used for Insulation Resistance Measurements

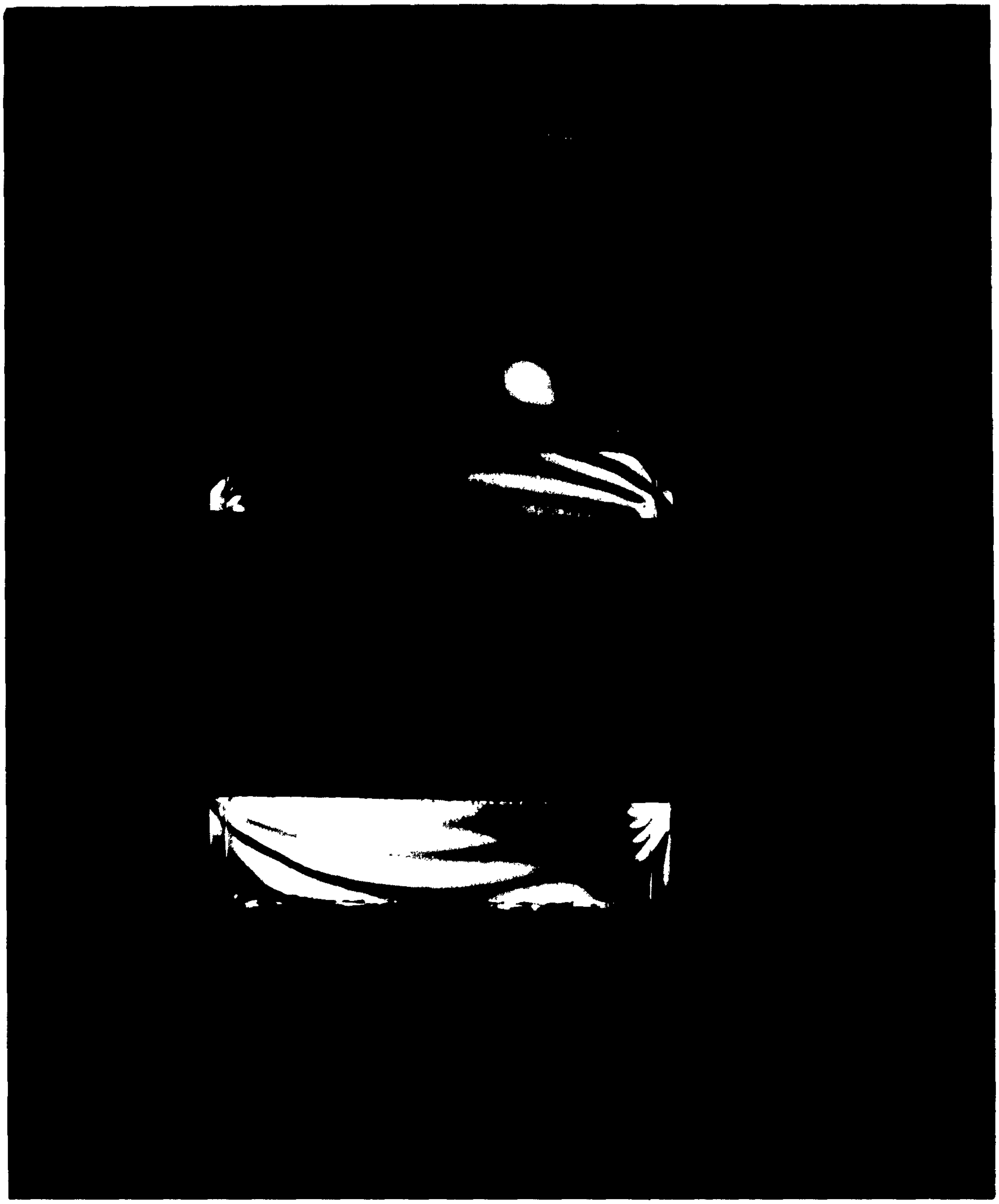

Fig. B-2 Strain Lines When Viewed With a Polariscope